



FIELD PARAMETER CERTIFICATION GUIDANCE DOCUMENT

**Office of Environmental Laboratory Certification
April 2010**

TABLE OF CONTENTS

	Page
STANDARD OPERATING PROCEDURES (SOP) MANUAL	3
CHAIN-OF-CUSTODY	5
SAMPLING PROCEDURES	6
PROFICIENCY TESTING (PT) SAMPLES	8
TEMPERATURE	11
TEMPERATURE CHECKLIST	12
HYDROGEN-ION CONCENTRATION (pH)	13
HYDROGEN-ION CONCENTRATION (pH) CHECKLIST	15
DISSOLVED OXYGEN (DO)	16
DISSOLVED OXYGEN (DO) CHECKLIST	18
RESIDUAL CHLORINE.....	20
RESIDUAL CHLORINE CHECKLIST	23
REFERENCES.....	25

APPENDICES

- I Sample Benchsheet for Thermometer Check Record
- II Sample Benchsheet for Hydrogen-Ion Concentration (pH)
- III Sample Benchsheet for Residual Chlorine
- IV Sample Residual Chlorine Initial Calibration Verification Form
- V. Certification Requirements for Laboratories Operating with Multiple Operators and/or Facilities
(Includes Field Parameter Laboratories)

STANDARD OPERATING PROCEDURES (SOP)s

Each certified laboratory must have a standard operating procedures (SOP) for each certified method.. The SOP must document the specific stepwise instructions for each parameter it is certified to perform, and must be readily available to each analyst for reference. SOPs must be reviewed and revised when changes are made in the laboratory procedures. Each SOP must include, but is not limited to the following information:

1. Revision Date
 Ensure that SOPs have assigned revision dates. When revisions are made to the SOP, list the revisions along with the date performed. This section will provide an ongoing history for the SOP modifications.
2. Method Identification and Description
 Identify the approved method number and description.
3. Scope and Application
 Documents the type of samples analyzed and analytes of interest.
4. Summary of the Method
 Lists the approved method reference and a brief explanation of the procedure.
5. Definitions
 Defines terms used in methodology (necessary for new or inexperienced analysts).
6. Interferences
 Includes physical or chemical properties of the sample, contaminants from glassware, reagents, and other apparatus.
6. Safety Apparatus and Procedures
 Lists any safety items needed to perform the analysis as well as any safety precautions needed.
8. Instrumentation and Equipment
 Lists all instrumentation and equipment needed to perform the procedure.
9. Reagents and Consumable Materials
 Includes manufacturer, catalog number, and any other pertinent information.
10. Sample Preservation and Handling
 Lists specific requirements for each type of sample collected and analyzed (NPDES, Drinking Water, and/or Solid and Hazardous Waste).
11. Calibration Procedures and Reagent Standardizations
 Includes the preparation of reagents and calibration standards along with the amounts and concentrations of standard material. Also include holding times and storage conditions for all reagents and standard material.
12. Procedure
 Details the entire procedure for analyzing samples and quality control samples. Includes all pertinent steps.
13. Quality Control
 Includes all laboratory quality control practices along with the frequency and acceptance criteria for QC samples.

14. Data Reduction, Validation, and Reporting
Describes data reduction, calculations, and reporting procedures.
15. Approved Analytical Methods and References (EPA or SM)
This must include all procedures needed for the analysis of this parameters.

CHAIN-OF-CUSTODY

Each certified laboratory must have written instructions for sample collection and handling in its standard operating procedures manual. To ensure sample integrity, it is required that an accurate written record (chain-of-custody) be available to trace the possession and handling of samples from the moment of collection through analysis and final disposition. This is referred to as chain-of-custody and is important to ensure sample integrity and will be used in the event of litigation involving the sample results.

Records must include the following:

1. Collection date and time.
2. Sample collector's signature.
3. Unique sample identification number (Assigned by the laboratory upon receipt).
4. Sample location and description.
5. Sample type - grab or composite (composite samples must have start/finish time and date for composite sampler).
6. Number of containers per analysis requested.
Must be traceable to the analysis.
7. Container type - glass or plastic.
8. Analyses required.
9. Preservatives used - H₂SO₄, NaOH, ice, etc.
Must be traceable to the sample container and analysis.
10. Program area - WW, DW, GW, etc.
11. Sample matrix - liquid, soil, waste, etc.
12. Transfer signatures w/dates and times for both relinquishment and laboratory receipt (the laboratory should indicate FEDEX, UPS, etc. in the "relinquished to" space if applicable).
13. Receipts maintained when shipped by common carrier (FEDEX, UPS, etc.).
14. Temperature upon receipt in the laboratory.

Chain-of-custody forms are not required for field analyses (pH, DO, residual chlorine, and temperature) or any other analyses performed at the collection site (such as specific conductance) since the analyses are performed at the same time that the samples are collected. The instrumentation (brand/model) must be traceable to the field measurements performed. Dates and times of analysis must also be traceable to field analysis results. If other samples are collected at the same time the field measurements are performed, procedures must be available to ensure proper sample collection. See Sampling Procedures.

SAMPLING PROCEDURES

The objective of sampling is to collect a representative sample sufficient in volume to be transported conveniently and handled in the laboratory. Valid test results depend on the following:

1. Using proper sampling, sample handling, and preservation techniques.
2. Properly identifying the collected samples and documenting their collection in permanent field records.
3. Maintaining sample chain-of-custody for compliance samples.
4. Protecting the collected samples by properly packing and transporting them to a laboratory for analysis.

Samples must always be collected according to the approach that is specified by the permit or in the sampling analysis plan.

Grab Samples

A grab sample is a discrete sample collected at a specific point and at a particular instant in time.

Grab sampling is:

1. Used to characterize the material at a particular instant in time.
2. Always associated with instantaneous water and wastewater flow data.
3. Conducted when the water or wastewater stream is not continuous (e.g. batch discharges or intermittent flow).
4. Conducted when the characteristics of the water or waste stream are known to be constant or considered for all practical purposes to be constant.
5. Conducted when the sample is to be analyzed for parameters whose characteristics are likely to change significantly with time, i.e. dissolved gases, bacteria, etc.
6. Conducted when the sample is to be collected for analysis of a parameter such as oil and grease where the compositing process could significantly affect the observed concentration relative to the true concentration.
7. Conducted when data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.

Composite Samples

A composite sample is usually associated with wastewater treatment facility sampling.

A time composite sample is one containing a minimum of eight equal-volume discrete samples taken at equal time intervals over the compositing period. The time composite definition is sometimes used where wastewater flows do not vary more than $\pm 15\%$ of the average daily flow rate.

A flow proportional composite sample is one containing a minimum of eight discrete samples with volumes proportional to the flow rate during the compositing period. Flow proportional sampling is usually conducted when the water or wastewater flows vary more than $\pm 15\%$ from the average daily flow rate.

Composite sampling is:

1. Used when average constituent or waste concentrations are of interest.
2. Always associated with continuous flow data.
3. Used when the wastewater stream is continuous.
4. Used when it is necessary to calculate mass per unit time loadings.

Time composite samples will be used where wastewater flows are constant or are considered for all practical purposes to be constant. A time composite sample will be collected continuously or with constant sample volume and a constant time interval between samples.

A flow proportional composite sample is used when wastewater flow is highly variable (greater than $\pm 15\%$ of the average daily flow). A flow proportional composite sample will be collected continuously and proportional to the wastestream flow, with constant sample volume and the time between samples proportional to wastestream flow, or with a constant time interval between samples and a sample volume proportional to flow at the time of sampling.

Composite samples must be maintained at $\leq 6^{\circ}\text{C}$ during the sampling period. Many facilities use refrigerated samplers. If a refrigerated sampling device is not utilized, ice must be replenished as needed to ensure that the sampling environment is maintained at $\leq 6^{\circ}\text{C}$ during the 24 hour sampling period. The temperature of the sampling environment must be documented at the time the sample is harvested.

Sample Preservation

Grab and composite samples must be preserved properly within 15 minutes of collection. The required containers, preservation techniques, and holding times for specific parameters are listed in Table II of the Federal Register (40CFR Part 136). The table includes several footnotes that must be followed for compliance sample collection and preservation.

Continuous Monitoring Devices

Some laboratories may be using continuous monitoring instruments for the measurement of sample pH or residual chlorine. The use of such instruments is approved for compliance monitoring as long as they are operating in accordance with the accepted EPA analysis criteria. Therefore, such instrumentation must be calibrated at least daily to ensure the accuracy of the reported values. Appropriate records of the daily calibrations must be kept to verify that the checks have been performed.

Many of the continuous monitoring instruments now on the market can easily be directly calibrated on a daily basis. Direct calibration of any instrument is always preferred. However, the Department acknowledges that direct daily calibration of some of the continuous monitoring devices currently marketed can be difficult, if not impossible.

As an alternative to direct daily calibration, it will be acceptable for a laboratory to compare a sample value from the continuous monitoring instrument to a sample value obtained from a benchtop instrument that has been properly calibrated each day. For residual chlorine analysis, the value obtained from the continuous monitor must agree within 10% of the value obtained from the benchtop instrument. For pH analysis, the value obtained from the continuous monitor must be within 0.2 pH unit of the value obtained from the benchtop instrument.

PROFICIENCY TESTING (PT) SAMPLES

South Carolina Regulation 61-81 specifies that each laboratory certified by the State of South Carolina which is reporting Clean Water, Safe Drinking Water, or Solid and Hazardous Waste Act data to this Department must report "Acceptable" results for a blind proficiency testing (PT) sample (if available) for each parameter method listed on its environmental laboratory certificate. This requirement must be met to initially be certified and then on an annual basis thereafter.

Once the laboratory is certified, it must successfully analyze a PT sample (obtained from an approved provider) for each parameter and method listed on its environmental laboratory certificate annually.

Please also note that if you fax, e-mail, or enter your results on-line, we strongly recommend that you also mail them via postal mail or contact the Provider to ensure that the e-mail, internet data entry, or fax was received in its entirety. There have been instances where the PT Provider did not receive the results and the laboratory was decertified.

Proficiency Testing (PT) samples are used to measure a laboratory's proficiency in analyzing a particular analyte by a specified method. The laboratory must participate in Water Supply (WS) and/or Water Pollution (WP) studies annually as required. WP studies are to be used to report PT samples for aqueous wastewater and Solid and Hazardous Waste Methods. The laboratory must obtain samples that are part of an official study obtained from an A2LA-approved provider. A list of approved PT providers may be found on the A2LA website at www.a2la.org. A list of required PT parameters for South Carolina can be located on our website at www.scdhec.gov/labcert.

PT Schedule

In order to maintain certification in South Carolina, acceptable PT sample results for the laboratory must be received by December 31st each year for the Water Supply (WS) and/or Water Pollution (WP) studies. This means that the study the laboratory participates in must begin in the calendar year and end in the calendar year with the results received in our office by December 31. The PT Provider must submit these studies to this Office. We cannot accept PT results faxed from laboratories. Also remember to document your EPA Lab Code on the results to your provider. See the information below on how to obtain an EPA Lab Code.

Studies received in January will not be accepted for meeting the annual PT requirement. Our Office will not be able to accept any PT study results after the calendar year (December 31) for instances where SC was not designated to receive a copy of the report. You must designate on the PT results submitted to the provider that SC is to receive a copy of the report when you submit your results. If the results are not received from the PT providers by December 31st, you will be decertified for the applicable parameters and you must reapply for certification. If we were designated to receive a report and there was an error on the PT provider's part, then we must receive a letter from the PT provider identifying the error that occurred.

All PT samples must be part of an official WS and/or WP study. Quick turn-around PT samples are not acceptable. The PT samples must be formulated according to the EPA Criteria Document published December 1998 and the NELAC Fields of Proficiency Testing (FoPT) tables. A2LA has approved PT Providers based on the new criteria. Some PT Providers are not certified for every parameter, so it is necessary to verify the PT provider's certificate which is available on the A2LA website.

Reporting Results to PT Provider

Results must be sent to the provider prior to the study close date. No late results will be accepted. Most providers make results available immediately following the study close date so that laboratories can grade their own data and order repeat samples without waiting three weeks for the final reports to be issued. Because many study results are released immediately, data reported to the PT Provider after the close of the study (even if the PT Provider accepts and grades the data) cannot be used for certification and will

be viewed as "Unacceptable" by this Office. Any changes to any of the data reported to the vendor will not be accepted for certification purposes. Be sure that the data is correctly reported when it is submitted.

When completing the reporting forms to be sent to the PT Provider, you must indicate that the results are to be sent directly to this Office. All of the PT Providers have the Office address and will send the reports here. If there are any questions regarding this, please contact the PT Provider to ensure that they have the Office address. The laboratory's EPA Lab Code and the State Lab ID number must also be included. Please be sure to include this information. If this information is not included, the results may be inadvertently credited to the wrong laboratory and you may not get credit for participating in a PT Study. If you do not know or are unsure of your EPA Lab Code and/or State Lab ID numbers, please contact us for assistance.

Correct Method Reporting

When completing the reporting forms, be sure to include the correct method number(s) being used. It is critical that the proper method number be referenced since PT samples are now required for each method for which you are certified. To ensure that you are reporting the correct method, review your certificate. If you are using a method that is not listed on your certificate, please contact this Office so that your certificate can be updated. If you do not report PT results for each method for which you are certified, you will lose certification for the unreported method(s). Results for one method cannot be substituted for another method. Although some wastewater and solid and hazardous waste methods may use the same technology, each method must still be reported. For example, if the laboratory is certified for EPA Method 624 and SW-846 Method 8260B or EPA Method 200.7 and SW-846 Method 6010C, both methods must be reported. This can be accomplished by reporting results for both methods individually in the same study or as a combination such as "EPA 624/8260B" or "EPA 200.7/6010C". The SW-846 Methods will not be accepted in lieu of the wastewater methods since there are different quality control requirements between the methods and since the wastewater methods are required for wastewater compliance reporting. Please also note that the revision letter for the SW-846 Methods must also be noted to ensure the PT sample was analyzed according to the current revision. The method must include the entire method reference as is written on your certificate. For instance, for pH, you must write the entire method such as SM 4500HB. Writing SM 4500, SM 4500B, SM 4500H, 4500H, or any other combination is not correct. If you have any questions about this, please contact this Office.

What if you have multiple labs?

If you are the laboratory director or employee for more than one laboratory, you must treat each laboratory independently. Samples must be obtained, analyzed, and reported by each individual laboratory. Individual laboratories are determined by their state laboratory ID number and associated certificate(s). If separate ID numbers (5 numerical digits) and certificates are issued by this Office, then separate PT samples are required. The PT sample reports must reference the proper laboratory ID numbers (State and EPA). It is not acceptable to report one result for any sample for two different laboratories. It is also not acceptable to split a sample between laboratories. If you work at two locations, you must analyze one sample at the first location using all of the equipment and reagents at the first location. You must then order and analyze another sample at the second location using the equipment and reagents at the second location.

All sample analyses must be recorded in the daily analysis records. This serves as the permanent laboratory record. Analysis results for the PT samples must be reported as if they are actual samples using the study you are participating in as the sample ID. Also keep a copy of the report that you send to the PT Provider for grading so that if problems arise, you will have a record of what you sent.

Solid and Hazardous Waste Proficiency Testing Samples

Laboratories are now required to analyze and report PT sample results for all applicable solid and hazardous waste aqueous parameters and/or methods. Laboratories will be required to use the WP PT study to report results for parameter and/or method they are certified to perform. Although some wastewater and solid and hazardous waste methods may use the same technology, each method must still be reported to ensure that the method required QC is met. For example, if the laboratory is certified

for Standard Method 4500HB and EPA Method 9040C or EPA Methods 120.1 and 9050A, both methods must be reported for the PT results. This can be accomplished by reporting results for both methods individually in the same study or as a combination such as "SM 4500HB/EPA 9040C" or "EPA 120.1/9050A". The SW-846 Methods will not be accepted in lieu of the wastewater methods since there are different quality control requirements between the methods and since the wastewater methods are required for wastewater compliance reporting. Please also note that the revision letter for the SW-846 Methods must also be noted to ensure the PT sample was analyzed according to the current revision.

EPA Lab Code

Each laboratory must use their assigned EPA Lab Code on all reported PT results submitted to their PT Provider. Without this EPA Lab Code we are unable to credit the PT results to the correct laboratory. The EPA Lab Codes are assigned by the USEPA. They must be contacted at the phone number provided below.

If you do not have an EPA Lab Code or do not remember your EPA Lab Code call, fax, or write:

Mr. Charles Feldman
US EPA, M/S 140
26 W. Martin Luther King Drive
Cincinnati, Ohio 45268
Phone (513) 569-7671: Fax (513) 569-7191

PT results reported without the correct EPA Lab Code may not be accepted by our office, since the results cannot be credited to appropriate facility or laboratory.

If there are any questions concerning the Proficiency Testing (PT) Requirements, please contact the Office of Environmental Laboratory Certification at (803) 896-0970.

Corrective Action Required for Unacceptable PT Results

Laboratories are now required to submit corrective action for all unacceptable PT results. The corrective action required will be a letter identifying why the failure occurred, what was done to resolve the problem, and the participation in another PT study with an acceptable performance. Letters of corrective action must be submitted within 30 days upon receipt of the final PT report. Acceptable results for any failed parameters must be received in our office by the PT provider by December 31st.

TEMPERATURE

Temperature measurements can be made with glass/mercury, glass/alcohol or dial bimetallic Celsius ($^{\circ}\text{C}$) thermometers (graduated in 0.1, 0.2, 0.5, or 1°C increments). The devices must be checked for accuracy using a NIST or NIST-traceable reference thermometer. Glass/mercury and glass/alcohol thermometers must be checked at least annually. Dial bimetallic and digital thermometers must be checked at least quarterly. NIST reference thermometers must be recalibrated every five years except for digital NIST reference thermometers which must be calibrated annually (including the probe and meter together).

When an infrared detection device is used to measure the temperature of samples, the device must be verified at least every six months using a NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient ($20\text{-}30^{\circ}\text{C}$), iced (4°C) and frozen (0 to -5°C). Each day of use a single check of the IR must be made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer. Agreement between the two must be within 0.5°C , or the device must be recalibrated.

If any field meters equipped with temperature sensors are used to make temperature measurements for compliance purposes, then they must also be checked for accuracy against a NIST or NIST-traceable reference thermometer at least annually.

Thermometer Accuracy Check Procedure

1. Immerse laboratory thermometer (or other temperature sensing device) into a sample at the normal range of use for the thermometer or device.
2. Immerse the reference thermometer into the sample.
3. Allow both thermometers to stabilize.
4. Record the readings from both thermometers and note the difference.
5. Document thermometer accuracy checks with the following information:
 - a) NIST reference thermometer I.D. #.
 - b) laboratory thermometer I.D. #.
 - c) date of accuracy check.
 - d) NIST reference thermometer reading.
 - e) laboratory thermometer reading.
 - f) temperature correction.
 - g) analyst's initials.
6. Each thermometer, upon being checked for accuracy must be tagged with the following information:
 - a) date of accuracy check.
 - b) initials of the analyst performing the accuracy check.
 - c) temperature difference from the reference thermometer - which must be taken into account each time a temperature measurement is made in order to get an accurate reading.

Sample Analysis Procedure

1. Collect sample and analyze immediately (as soon as possible, not to exceed 15 minutes).
2. Immerse thermometer in sample.
3. Allow thermometer to stabilize.

4. Note temperature reading and compensate for any difference with the reference thermometer.
5. Record corrected temperature reading.

TEMPERATURE CHECKLIST

1. Type of thermometers/temperature sensors used:
 - a)
 - b)
 - c)
2. Source of NIST/NIST-traceable reference thermometer:
3. Is certificate for reference thermometer available?
4. Accuracy check records for thermometers and temperature sensors used?

Records include:

 - a) date.
 - b) reference thermometer number.
 - c) reading of reference thermometer.
 - d) identification of thermometer checked.
 - e) reading of thermometer checked.
 - f) difference/adjustment.
 - g) comments.
 - h) initials.
5. Thermometers/temperature sensors labeled with
 - a) date checked.
 - b) analyst.
 - c) correction value/adjustments.
6. Correction value/adjustment taken into account?

HYDROGEN-ION CONCENTRATION (pH)

Equipment and Supplies

1. A pH meter capable of reading pH values of a solution ranging from 0 - 14 standard units (SU), in 0.1 SU increments, with the appropriate electrode. The meter's accuracy must be no less than ± 0.1 SU.
2. Reference buffers of pH 7.0 and 4.0 or 10.0. Other standard pH solutions may be acceptable.
3. Deionized or distilled water, preferably in a squeeze bottle.
4. Small beakers (glass or teflon) for holding buffer solutions during calibrations.
5. Laboratory benchsheet for documentation of calibration and analysis.
6. Optional:
 - a) magnetic stirring bar.
 - b) magnetic stirring plate - please be aware that the stirring plate can generate heat over an extended period of time and change the temperature of buffer solutions and samples.
 - c) lint free tissues, such as Kim-Wipes - as recommended by manufacturer

Calibration Criteria

1. The instrument must be calibrated at two points using two different buffers. The pH 7.0 buffer must be used and the laboratory must select either a pH 4.0 or pH 10.0 buffer for the second point. The two buffers chosen must bracket the expected pH value of the sample to be analyzed as closely as possible. For example, if the analyst expects the pH of the sample to be 7.9, then pH 7.0 and 10.0 buffers should be used for the calibration.
2. It is recommended that a third buffer be used to check the accuracy of the meter. A three-point calibration can be performed.
3. Each work shift the meter must be recalibrated.
4. For facilities that have to perform field analysis at multiple sites, the pH meter can be initially calibrated in the laboratory and then checked with a pH 7.0 buffer at each site before sample analysis.
5. Buffers must not be used more than once. At each calibration, a fresh aliquot of buffer must be used.
6. Documentation of all calibrations must be maintained and must be readily available for examination. The records must, at a minimum, include the following information:
 - a) date of calibration.
 - b) time of calibration.
 - c) initials of analyst.
 - d) meter I.D. (if more than one meter is used by the laboratory).
 - e) reading for the pH 7.0 buffer after meter adjustment.
 - f) reading for the pH 4.0 or 10.0 buffer after meter adjustment.
 - g) slope and/or efficiency reading if given by the meter (the slope/efficiency reading is used as an indicator of electrode problems)
 - h) comment section.

7. Buffers must not be used after their expiration dates, as provided on the container by the manufacturer. If the manufacturer fails to provide an expiration date, the laboratory must assign an expiration date of one year after opening in the laboratory. The date received in the laboratory, the date opened, and the expiration date must be recorded on all containers of pH buffer.
8. When buffers are transferred to a secondary container for temporary storage, the lot #, buffer pH value, expiration date, and analyst's initials must be written on the container.
9. Buffer solutions prepared by the laboratory from pH powder pillows or liquid concentrates can be used up to one year from the date that they are prepared. The container must be labeled with the date prepared, lot # of the pillows, buffer pH value, expiration date, and preparer's initials.
10. The instrument must be calibrated each day (per work shift) before it is used. When the tip of the electrode is submerged in the buffer during calibrations, gently swirl it until the meter reading stabilizes. Alternatively, a magnetic stirring bar and stirrer can be used.
11. When transferring the electrode from one buffer to another, the electrode must be rinsed with deionized or distilled water and gently wiped or blotted with a lint-free tissue (unless discouraged by the manufacturer).
12. The electrode must be stored and maintained according to the manufacturer's instructions. This information must be included in the SOP.
13. Document any maintenance performed on the meter or the electrode. Meter must be recalibrated before use after any maintenance.
14. If the instrument is equipped with an automatic temperature compensator (ATC), it is recommended that the device be checked for accuracy with a NIST or NIST-traceable reference thermometer. See TEMPERATURE section of this document for details. If the instrument is used for reporting temperature, the sensor must be checked for accuracy annually.

Analysis Criteria

1. The pH meter must be calibrated (each work shift) before any analyses are performed.
2. The analyst must maintain documentation of all analyses. The records must include, at a minimum, the following information:
 - a) date of analysis
 - b) time of sample collection and time of sample analysis
 - c) analyst's initials
 - d) instrument ID (if more than one meter is used by the laboratory)
 - e) sample identification
 - f) pH value of sample
 - g) comment/maintenance section
3. If more than one sample is analyzed in a series, the electrode must be rinsed with deionized or distilled water and blotted gently with a lint-free tissue (unless discouraged by the manufacturer) between samples.
4. When analysis is complete, the electrode must be rinsed and stored according to the manufacturer's instructions.
5. Instrument maintenance must be performed as required by the manufacturer.

HYDROGEN-ION CONCENTRATION (pH) CHECKLIST

1. Manufacturer:
2. Instrument and electrode clean?
3. Buffers within expiration dates? 7 ___ 4 ___ 10 ___
4. Electrode:
 - a) storage solution.
 - c) if nonsealed, filled to proper level with electrolyte solution?
5. Buffer aliquots changed daily/used only once?
6. Deionized/distilled water in squeeze bottle, used to rinse electrode between samples and buffers?
7. If buffers are transferred to secondary containers for use and storage, document the pH value, expiration date, analyst's initials, and lot number on secondary containers?
8. Instrument calibrated before use each work shift?

Calibration records must include:

 - a) date
 - b) time
 - c) analyst
 - d) instrument identification (if more than one instrument is used)
 - e) Documentation of the pH 7 buffer checks
 - f) Documentation of the pH 4 and/or 10 buffer checks*
*Buffers must bracket sample values.
 - g) third buffer check (optional - highly recommended)
 - h) slope or efficiency (if given by instrument)
 - i) comments/maintenance
9. Sample analysis records must include:
 - a) date
 - b) time sample is collected
 - c) time sample is analyzed
 - d) analyst's initials
 - e) instrument ID (if more than one instrument is used)
 - f) sample identification
 - g) pH value
 - h) comments/maintenance
10. Is sample analyzed within 15 minutes of collection?
11. Is sample stirred gently during analysis?

DISSOLVED OXYGEN (DO) – Membrane Electrode Method and Luminescence Method

Equipment and Supplies

1. An oxygen-sensitive membrane probe or LDO probe and the appropriate meter
2. Laboratory benchsheet for documentation of calibration and analysis

Calibration Criteria

1. The instrument must be calibrated each day (per work shift) before it is used.
2. The instrument must be calibrated according to the manufacturer's instructions.
3. Documentation of all calibrations must be maintained by the laboratory and must be readily available for review. Calibration records must reflect the calibration procedure required by the manufacturer. The records must, at a minimum, include the following information provided it is applicable to the instrument in use:
 - a) date and time of calibration.
 - b) meter ID, if more than one meter is used by the laboratory
 - c) analyst's initials
 - d) membrane check
 - e) comment/maintenance section
 - f) adjusted/final DO value

Depending on the type of meter in use, the following information must be recorded. If the laboratory's meter provides or requires the following, it must be recorded. If the meter does not provide or require the following, the information is not required.

- g) air temperature
- h) initial DO value
- i) zero check
- j) red-line check
- k) altitude adjustment
- l) battery check

There are also several meters that have an auto calibration mode. The membrane probe is placed inside a calibration chamber that has a 100% water saturated air environment. Typically, these meters require the operator to input the local altitude or barometric pressure and the salinity of the water to be analyzed. For these types of DO meters, the following information must be recorded.

- m) altitude
- n) barometric pressure
- o) temperature
- p) salinity
- q) calibrated or final DO reading
- r) % saturation

For luminescent dissolved oxygen (LDO) meters, the following calibration information must also be recorded:

- s) membrane cap ID and date of expiration
- t) theoretical DO value, as obtained from DO tables and corrected for your altitude/pressure

- u) DO reading of blank (either water-saturated air or air-saturated water) to compare with the theoretical value

Note that the blank DO value must read within 97-104% of the theoretical DO value for your altitude/pressure.

4. The DO probe must be stored according to the manufacturer's instructions.
5. The DO probe's temperature sensor must be checked for accuracy at least annually using a NIST or NIST-traceable reference thermometer. This is required even if the meter is not used for reporting temperature. This check must be recorded on a thermometer check log. The probe must then be tagged with the following information:
 - a) date of temperature check
 - b) initials of analyst performing the accuracy check
 - c) temperature difference between the probe and the NIST-traceable thermometer
6. If the temperature difference from the reference thermometer is more than 1.0°C, then the probe must be repaired or replaced.
7. Records of maintenance performed on the instrument must be kept for review (e.g. membrane, battery, and probe changes). This information can be placed in the comments section of the sample analysis record.

Analysis Records

1. The instrument must be calibrated before any sample analyses can be performed.
2. The analysis records must include, at a minimum, the following information:
 - a) date of analysis
 - b) time of sample collection and sample analysis
 - c) sample I.D. (name or number)
 - d) dissolved oxygen (DO) reading in mg/L (ppm)
 - e) analyst's initials
 - f) comments
 - g) instrument ID if more than one instrument is used in the laboratory
 - h) LCS/LCS duplicate analyses (LDO only). The LCS/LCS duplicate samples are water-saturated air (or air-saturated water) samples that must be analyzed with each sample batch and read within 97-104% of each other and the theoretical DO value.

DISSOLVED OXYGEN (DO) CONCENTRATION CHECKLIST

1. Manufacturer:
2. Instrument and probe clean?
3. Membrane in good condition?
Wrinkles, tears, air bubbles?
4. How is probe stored?

When not in use -
During saturated air calibration -
5. Temperature sensor checked for accuracy?
6. Instrument calibrated (per work shift) before use?
Calibration records include:
 - a) date and time of calibration
 - b) meter number if more than one meter is used by the laboratory
 - c) analyst's initials
 - d) membrane check
 - e) comment/maintenance section
 - f) adjusted DO value

Depending on the type of meter in use, the following information must be recorded. If the laboratory's meter provides or requires the following, it must be recorded. If the meter does not provide or require the following, the information is not required.

- g) air temperature
- h) initial DO value
- i) zero check
- j) red-line check
- k) altitude adjustment
- l) battery check

There are also several meters that have an auto calibration mode. The membrane probe is placed inside a calibration chamber that has a 100% water saturated air environment. Typically, these meters require the operator to input the local altitude or barometric pressure and the salinity of the water to be analyzed. For these types of DO meters, the following information must be recorded.

- m) altitude
- n) barometric pressure
- o) temperature
- p) salinity
- q) calibrated or final DO reading
- r) % saturation

For luminescent dissolved oxygen (LDO) meters, the following calibration information must also be recorded:

- s) membrane cap ID and date of expiration
- t) theoretical DO value, as obtained from DO tables and corrected for your altitude/pressure
- u) DO reading of blank (either water-saturated air or air-saturated water) to compare with the theoretical value

Note that the blank DO value must read within 97-104% of the theoretical DO value for your altitude/pressure.

7. Sample analysis records must include:
 - a) date of analysis.
 - b) time of sample collection and sample analysis
 - c) sample I.D. (name or number)
 - d) dissolved oxygen (DO) reading in mg/L (ppm)
 - e) analyst's initials
 - f) comments
 - g) Instrument ID, if more than one instrument is used in the laboratory
 - h) LCS/LCS duplicate analyses (LDO only). The LCS/LCS duplicate samples are water-saturated air (or air-saturated water) samples that must be analyzed with each sample batch and read within 97-104% of each other and the theoretical DO value.
8. Is sample analyzed within 15 minutes?
9. Is sample stirred gently during analysis?

RESIDUAL CHLORINE – DPD Spectrophotometric or Colorimetric

Equipment and Supplies

1. A spectrophotometer or colorimeter, for use at an appropriate wavelength and providing a light path of 1 cm or longer
2. Spectrophotometer cells (vials)
3. DPD reagent powder pillows (free or total)
4. Volumetric glassware to prepare standards
 - a) volumetric pipets
 - b) volumetric flasks
5. Potassium permanganate (KMnO_4) or commercially prepared chlorine stock standard solutions may be used
 - a) Potassium permanganate (KMnO_4) primary stock solution (1000ppm chlorine equivalent) - 0.891g KMnO_4 /L
 - 1) made fresh at least every 6 months
 - 2) stored in a glass container protected from light and kept in cool, dry place
 - 3) labeled with date prepared, reagent name and concentration, and analyst's initials
 - b) Commercially prepared KMnO_4 primary stock solution (1000ppm chlorine equivalent) - 0.891g KMnO_4 /L
 - 1) purchased in an amber glass container
 - 2) unopened container good up to the manufacturer's expiration date
 - 3) shelf-life of six months once opened*
*The six month shelf-life will take precedence over any extended expiration date that the manufacturer may have assigned but cannot exceed the manufacturer's expiration date, if provided.
 - 4) once opened, labeled with date opened, six month expiration date, and analyst's initials
 - 5) stored in a cool, dry place
 - c) Secondary stock solution (100ppm chlorine equivalent) - dilute 10 ml of primary stock to 100 ml
 - 1) made fresh at least every week
 - 2) stored in a glass container protected from light and kept in a cool, dry place
 - d) Working standards
 - 1) made fresh daily (per work shift) in glass containers
 - 2) a minimum of two concentrations used daily (per work shift)

If degradation is observed or standard readings differ more than 10% from the assigned standard values, new secondary and/or new primary standard solutions must be made before the normal expiration dates.

6. Commercially prepared liquid chlorine standard solution must be used according to the manufacturer's instructions and diluted to prepare working standards. Gel standards are not approved for use.

Initial Calibration Verification

1. An initial calibration verification must be performed annually or when a new lot number of DPD reagent pillows is used, whichever is sooner.
2. A separate initial calibration verification must be generated for DPD free and total reagent powder pillows.
 - a) a separate calibration verification must be generated for each type of sample vial (2.5 cm and 1.0 cm, other) used
3. A minimum of 5 standards and a blank covering the range of the instrument's capabilities must be used to generate each initial calibration verification.
4. Each initial calibration verification must, at a minimum, be labeled with the following information:
 - a) date and time generated
 - b) analyst's initials
 - c) instrument I.D. number (if more than one instrument is used by the laboratory)
 - d) lot number of DPD reagent pillows
 - e) cell path length (if interchangeable)
 - f) true concentration of standards
 - g) observed concentration (meter readings) or absorbance of standards
 - h) wavelength (if adjustable)
 - i) record of true standard concentration vs. observed standard concentrations. All observed (meter readings) must be within 10% of the true standard concentration. If not, prepare fresh standards. If the 10% criteria still cannot be met, contact the manufacturer for service. (Note: For the 0.05 mg/L standard, the acceptable range is 80-120% because most colorimeters are capable of displaying only two decimal places.)
 - j) identification of results as total or free residual chlorine

Daily Calibration Verification Criteria

1. The instrument calibration must be verified daily (per work shift) before it is used.
2. A minimum of two standards and a blank must be used for daily (per work shift) calibration verification. The observed concentration for each standard must be within 10% of its true concentration.
3. The daily calibration verification records must be readily available for examination and must, at a minimum, include the following information:
 - a) date of calibration
 - b) time of calibration
 - c) instrument I.D. number if more than one is used in the laboratory
 - d) blank analysis
 - e) true concentration of standards
 - f) observed concentration for each standard. The observed value must be within 10% of the true value.
 - g) lot number of DPD reagent pillows (must be traceable to the initial calibration record.)

- h) initials of analyst
- i) wavelength (if adjustable)
- j) cell path length (if interchangeable)
- k) identification of results as total or free chlorine

Analysis Criteria

1. The instrument's calibration must be verified each work shift before conducting sample analysis.
2. Documentation of all analyses must be readily available for examination and must, at a minimum, include the following information:
 - a) date of analysis
 - b) time of analysis (samples must be analyzed within 15 minutes after collection)
 - c) analyst's initials
 - d) instrument used (if more than one is used in the laboratory)
 - e) sample identification
 - f) sample chlorine value, mg/L (ppm)
 - g) lot number of DPD reagent pillows. (Must be traceable to the initial and daily calibration verification.)
 - h) comment section
 - i) Identification of results as total or free chlorine
3. When reporting results, the laboratory cannot report values that are lower than the lowest standard concentration used to generate the initial calibration verification. For example, if the lowest standard used to perform the initial calibration verification is 0.1 mg/L, the minimum reporting limit is 0.1 mg/L. If the meter reads less than 0.1 mg/L, then the analyst should report the value as "<0.1 mg/L". Daily calibration verifications can be performed with higher or different standard concentrations. For example, 0.5 and 1.0 mg/L standards can be used for daily calibrations.

TOTAL RESIDUAL CHLORINE CHECKLIST

1. Instrument:
Instrument and equipment (vials) clean:
2. SOP manual
3. Initial Calibration Verification
 - a) Generated annually or when lab uses a new lot of DPD pillows
 - b) Separate calibration verification required for total and free residual chlorine
 - c) Use 5 standards and blank covering range of meter capabilities
 - d) Labeled with:
 - 1) date generated and time generated
 - 2) analyst's initials
 - 3) instrument I.D. (if more than one instrument is used in the laboratory)
 - 4) lot number of DPD reagent pillows
 - 5) cell path length (if adjustable)
 - 6) true concentration of standards
 - 7) observed concentration (meter reading) of standards
 - 8) wavelength (if adjustable)
 - 9) record true concentration vs. observed (meter reading) concentration . All observed (meter readings) must be within 10% of the true standard concentration. If not, prepare fresh standards. If the 10% criteria still cannot be met, send meter for service. (Note: For the 0.05 mg/L standard, the acceptable range is 80-120% because most colorimeters are capable of displaying to only two decimal places.)
 - 10) identification as total or free chlorine
4. Stock solutions and standards
 - a) Primary stock solution (1000ppm chlorine equivalent) - 0.891g/L potassium permanganate
 - 1) made fresh at least every 6 months
 - 2) stored in a cool, dry place
 - 3) stored in a glass container protected from light
 - 4) labeled with date prepared and initials of analyst
 - b) Commercially prepared KMnO_4 primary stock solution (1000ppm chlorine equivalent) - 0.891g KMnO_4 /L
 - 1) purchased in an amber glass container
 - 2) unopened container within the manufacturer's expiration date
 - 3) opened containers within 6 month holding time*
 - 4) stored in a cool, dry place and in a glass container
 - 5) once opened: labeled with date opened, six month expiration date, and analyst's initials

*The six month shelf-life will take precedence over any extended expiration date that the manufacturer may have assigned but may not exceed the manufacturer's expiration date.
 - c) Secondary stock solution (100ppm chlorine equivalent)
 - 1) 10 ml of 1000ppm stock to 100 ml
 - 2) made fresh weekly

- 3) stored in a glass container and in a cool, dry place
 - 4) stored protected from light
- d) Commercially Prepared Chlorine Standards
 - 1) used according to manufacturer's instructions
 - 2) diluted to prepare working standards
- e) Calibration Verification Working Standards
 - 1) Initial Calibration Verification - five concentrations covering the range of the analyses
 - 2) Daily Calibration Verification - two concentrations in working range
 - 3) Both initial and daily calibration working standards made fresh daily(per work shift)
- f) Volumetric glassware used to prepare standards
 - 1) volumetric pipets
 - 2) volumetric flasks
- g) Daily Calibration Verification
 - 1) Calibration verification performed daily, before use of instrument
 - 2) Use of two standards and a blank
 - 3) Calibration records include:
 - a) date of calibration
 - b) time of calibration
 - c) instrument I.D. (if more than one instrument is used by the laboratory)
 - d) lot number of DPD reagent pillows (must be traceable to the initial calibration)
 - e) true concentration of standards
 - f) observed (meter reading) concentration of standards. (must be within 10% of the true value)
 - g) analyst's initials
 - h) identification of results as total or free chlorine
- h) Analysis records include:
 - 1) date
 - 2) time sample collected and analyzed
 - 3) instrument I.D. (if more than one instrument is used by the laboratory)
 - 4) sample reading, mg/L (ppm)
 - 5) analyst's initials
 - 6) lot number of DPD reagent pillows (must be traceable to the initial calibration and daily calibration)
 - 7) identification of results as total or free chlorine
- j) Maintenance records
 - 1) batteries replaced.
 - 2) service documented.

REFERENCES

1. Standard Methods for the Examination of Water and Wastewater, 18th, 19th, 20th, and 21st Edition.
American Public Health Association.
2. 40 CFR Parts 141 and 142 for Safe Drinking Water Act.
3. 40 CFR Part 136 for Clean Water Act.

THERMOMETER CHECK RECORD

- A. National Institute of Standards and Technology (NIST) Certified Thermometer or equivalent thermometer identification number: _____
- B. Identification number of thermometer that is being calibrated, identified as "B".
Thermometers must be checked in the range of use. For example: If the thermometer is used in the refrigerator to monitor 0-4°C, the thermometer must be checked against the NIST-traceable thermometer at 4°C.

[illegible]

Laboratory Name _____

Hydrogen-Ion Concentration (pH)

Meter Model/I.D. _____

Year: _____

Calibration		CALIBRATION				SAMPLE ANALYSIS		BOTH			
Date	Time	pH 7 Buffer	pH 4 Buffer	pH 10 Buffer	Slope or Efficiency % or mV	Time** Collected	Time** Analyzed	Sample ID (name or number)	Sample Result	Analyst's Initials	Comments

** Must meet the 15-minute holding time between sample collection and analysis

Laboratory Name: _____

***Total /Free Residual Chlorine**

DPD lot # _____

Year: _____

Meter/Model# _____

Initial Calibration Verification Date_____

-----DAILY CALIBRATION-----|-----SAMPLE ANALYSIS-----|-----BOTH-----

[illegible]

*Indicate whether the analyses are total residual chlorine or free residual chlorine by circling the Total or Free at the top of the page.

**Daily meter readings for the standards must be within 10% of the true value of the standards. Please record the true value of the standard here.

***15 minute holding time between collection and analysis

Sample Residual Chlorine Initial Calibration Verification Record

Date/Time: _____

Instrument ID: _____

Analyst: _____

DPD Lot#: _____

Cell Path Length: _____

*Wavelength: _____

Total/Free Residual Chlorine (Circle the type of residual chlorine recorded)

True Value of Standards mg/L	*Observed (Meter Readings) mg/L
Blank	
Std 1 =	
Std 2 =	
Std 3 =	
Std 4 =	
Std 5 =	

* Only required to be recorded if the meter wavelength is adjustable on the instrument used for analysis.

** The lowest standard on the initial calibration verification is the Practical Quantitation Limit (PQL). This is used for reporting. For example: The lowest non-zero standard on the initial calibration verification is 0.05mg/L. The laboratory obtains a sample reading of 0.02mg/L. The laboratory would record the sample value as <0.05mg/L on the sample analysis record since 0.05mg/L is the lowest standard on the initial calibration verification.

*** The observed readings must be within 10% of the true value. If the meter readings are different by more than 10%, maintenance must be performed. An acceptance range of 80-120% is permitted for the 0.05 mg/L standard because most colorimeters display results to only two decimal places. Maintenance includes cleaning the sample cells, remaking the standard, and possibly returning to the manufacturer for repair.



**Certification Requirements for Laboratories Operating
With Multiple Operators and/or Facilities
(Includes Field Parameter Laboratories)**

When multiple operators/facilities are reporting regulatory analyses under a single SC Laboratory I.D. number, the following procedures must be followed:

The laboratory must designate one person as the Laboratory Director. This person must assume the responsibility of ensuring that the following criteria are being met:

- a) The Laboratory Director must be given the responsibility by the laboratory's governing body (owners, directors, commissioners, councilmen, mayor, board members, or who-so-ever occupies the status of proprietor) of supervising the operations of all the operators/facilities and ensuring the quality of data reported on a daily basis.
- b) All laboratory technicians/operators performing regulatory analyses have been adequately trained to calibrate and use all pertinent instruments. Training records must be available for review.
- c) All lab technicians/operators report for work each day at the certified laboratory location where the equipment, reagents, and records are being stored, to calibrate their equipment before going to their specific facility. A complete listing of personnel approved to report under the SC laboratory ID number must be maintained on file at the certified location.
- d) Satisfy the proficiency testing (PT) requirements as documented in the SCDHEC "Proficiency Testing (PT) Requirements". It is also recommended that an unknown sample be analyzed annually by all lab technicians/operators and placed on their training file as part of their Quality System.

On-Site Evaluations

When multiple operators/facilities of the same corporate body prefer to report under one SC Laboratory Identification (I.D.) number, the on-site evaluation process initiated by this Office will be to conduct the on-site evaluation for all entities associated with the SC Laboratory I.D. number at the certified laboratory location where the equipment, reagents, and records are being stored. All equipment, analysis records, and quality control records for each operator/facility must be at this location for review. Each operator/facility must be represented at the evaluation.

When is a separate SC Laboratory Identification (I.D.) Number needed for multiple operators and/or facilities?

If an entity with multiple operators/facilities chooses not to or is unable to comply with items a-d above, then certification will be required for each operator/facility and separate on-site evaluations will be performed for each operator/facility. A SC Laboratory I.D. Number will be assigned to each certified operator/facility upon successful completion of the certification application process.

The following criteria must be met if separate SC Laboratory I.D. numbers are assigned:

- a) A complete application package with a \$125 application fee must be submitted for each operator/facility.
- b) Annually, a proficiency testing (PT) sample must be analyzed by each operator/facility for each certified parameter to satisfy the proficiency testing (PT) requirements as documented in the "SCDHEC Proficiency Testing (PT) Requirements". "Acceptable" results must be achieved. An EPA Lab Code must be received from the EPA for reporting results to the Proficiency Testing Provider.

- c) Each operator/facility will be considered a unique laboratory and will be given a separate SC Laboratory I.D. Number. Each operator and/or facility must have standard operating procedures and a list of personnel operating under this SC Laboratory I.D. Number along with pertinent training records.

Invoicing/Billing Information

Annually, each operator/facility issued a unique SC Laboratory I.D. Number will receive a separate invoice for the applicable laboratory certification fees.